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DECONTAMINATION TESTS ON STAINLESS STEEL TUBING REMOVED FROM THE DARLINGTON TRITIUM REMOVAL FACILITY.

Report No 90-100-K

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ABSTRACT

Modifications to process tubing in the High Tritium Columns associated with the Darlington Tritium Removal Facility (TRF) are necessary to permit reliable determination of liquid tritium levels. Before these modifications could be undertaken, samples of tubing with similar tritium exposure history were characterized with respect to total tritium inventory, amount of HTO relative to HT and the amount of smearable tritium activity, in order to determine the effect on the working environment when the actual field tubing is cut and welded. The thermal desorption tests carried out demonstrated that high airborne tritium levels, mainly as HTO, of the order of 2500 MPC_a would be produced during the modifications unless good ventilation of the working area was assured. In addition to providing information to Operations for their planning of the TRF modifications, the work provided some insight into developing decontamination techniques for tritium contaminated surfaces.

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EXECUTIVE SUMMARY

DECONTAMINATION TESTS ON STAINLESS STEEL TUBING FROM THE DARLINGTON TRITIUM REMOVAL FACILITY

J.P. Krasznai and W.T. Shmayda

Modifications to process tubing in the High Tritium Columns associated with the Darlington Tritium Removal Facility are necessary to permit reliable determination of liquid tritium levels. Concerns were raised by the mechanics involved with these modifications that the high tritium surface contamination levels on the process tubing could result in very high concentrations of airborne tritium when subjected to welding torch temperatures.

To determine the extent of this problem samples of tubing similar in tritium exposure history to the tubing to be cut and welded were characterized at the Research Division Laboratory. The tritium inventory as HTO and HT was determined as well as the rate of release of these species as a function of temperature. In one experiment the airborne tritium concentration was measured when a sample of tubing was subjected to an instantaneous temperature increase of 1000°C to simulate a welding torch.

The results of our investigations indicated that the tritium surface activity of the tubing is 0.33 mCi/cm^2. The predominant tritium species released is HTO while the HT component is less than 5% of the total. The quantity of smearable tritium contamination (1.3 mCi) was 26% of the total tritium activity.

Thermal desorption of tritium at 350°C was an effective technique for the decontamination of surface tritium from stainless steel surfaces. This temperature will not adversely affect the mechanical or metallurgical integrity of the tubing.

The airborne tritium concentration in an unventilated 2 m^3 plastic tent generated by mechanically cutting, and then heating a 30 cm length of tubing is approximately 2500 MPC_as assuming an MPC_a value of 10 uCi/m^3 for HTO and also assuming that all of the desorbed tritium escapes into the tent atmosphere. Clearly, good ventilation of the tent atmosphere is of paramount importance.

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The experiments carried out in this work were designed to answer very specific concerns regarding tritium surface contamination required to be resolved for work planning purposes. In order to develop effective decontamination techniques for a variety of expected tritium contaminated surfaces it will be necessary to undertake additional systematic studies to:

(a) characterize the nature of tritium surface contamination on different surfaces, metallic as well as non metallic including different grades of stainless steel as a function of tritium partial pressure and exposure time,

(b) determine the thermal desorption characteristics of the tritium species on the various substrates to assess the nature of bonding of tritium to the surfaces,

(c) investigate various tritium decontamination techniques in addition to thermal desorption such as photon induced desorption and electrolytic methods and

(d) assess the rate of recontamination of a decontaminated surface in both a static and flowing gas environment.

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DECONTAMINATION TESTS ON STAINLESS STEEL TUBING FROM THE DARLINGTON TRITIUM REMOVAL FACILITY

J.P. Krasznai and W.T. Shmayda

INTRODUCTION

The Darlington Tritium Removal Facility is designed to extract tritium from heavy water. The system consists of a complex system of stainless steel tubing carrying hydrogen isotopes in both gaseous and liquid forms.

There has been a problem measuring liquid tritium levels in the High Tritium Columns. This will be resolved by replacing part of the High Tritium Column 1 with larger bore tubing. As with all tubing carrying tritium, the joints must be welded. There was a concern expressed by the mechanics responsible for the repairs that tritium surface contamination in the tubing to be replaced would be converted to very high levels of airborne tritium, estimated at several hundred thousand MPC_as during the welding operation.

To assist the Darlington Technical Unit develop the work plans for the repairs, Research Division was requested:

(a) to determine the tritium specific activity in a representative sample of tubing,

(b) to identify the nature of the tritium species on the metal surface,

(c) to determine whether decontamination prior to cutting and welding is required and if so,

(d) to identify the optimum decontamination procedures.

This report summarizes the work carried out jointly by the Materials Chemistry Section of the Chemical Research Department and the Corrosion and Tritium Technology Section of the Metallurgical Research Department.

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EXPERIMENTAL

The equipment used in this work is shown schematically in Figure 1. In the first tube furnace consisting of a heated quartz tube the sample is heated to drive off the tritium. The carrier gas, a mixture of nitrogen (200 mL/min) and hydrogen (10 mL/min), carried the tritium through two bubblers in series containing ethylene glycol and water to trap any exchangeable hydrogen species such as HTO. Any non exchangeable hydrogen species such as HT or organic species containing tritium would be oxidized in the second tube furnace containing copper oxide heated to 750°C and subsequently trapped in the second series of bubblers.

A low flow of hydrogen gas is maintained in order to minimize the amount of tritium adsorbed onto the walls of the equipment following desorption from the steel. The product of oxidation of HT or the other tritiated organics is essentially HTO which is trapped in the second set of bubblers at the outlet of the tube furnace. Gas samples could be withdrawn via hyperdermic sample ports at the inlet of the first and second set of bubblers. All tritium determinations were done using liquid scintillation counting.

Three experiments were carried out. In the first a small sample (3cm long 3/8" diameter tube) was heated sequentially from 100°C to 1000°C over an eight hour period and the desorbed tritiated species were determined as a function of both time and temperature.

The second experiment was essentially a duplicate of the first except a temperature that was both easily achievable at Darlington and not likely to adversely affect the metallurgical properties of stainless steel was selected. The second sample of the tubing, 3.6cm long, was heated at 350°C for 4 hours and the total tritium as HTO and HT was determined. Subsequently the temperature was increased to 1000°C for an additional 4 hours to confirm whether all the tritium associated with the specimen had in fact been removed.

The objective of the third experiment was to simulate the weld conditions and measure the tritium in air concentration. This was achieved by connecting a high range Scintrex meter to the outlet of the desorption furnace which was pre-heated to 1000°C and then quickly introducing a sample of the stainless steel tubing into the furnace.

RESULTS AND DISCUSSION

In the first experiment the tritium content of the bubblers, was monitored, Table 1, over an 8 hour period of desorption at high temperature. Bubbler B1,B2 and B3,B4 represent the total HTO and HT removed during the experiment respectively.

The total tritium inventory in the steel specimen was 3.73 mCi, 97% of which was in a form readily exchangeable with water and assumed to be HTO. The external surface of the tubing had been exposed to tritium gas. A smear was initially taken and the loose tritium contamination was determined by liquid scintillation counting. The measured activity on the smear was 0.15 mCi/cm^2 and a total of 1.3 mCi was removed. Therefore adding the smearable activity to that desorbed thermally and assuming that the degree of internal and external contamination are equal, gives a total surface activity of 0.33 mCi/cm^2. This is 6700 times the maximum permissible limit (MPL) set by Ontario Hydro for tritium surface contamination present as HTO.

TABLE 1

EFFECT OF TEMPERATURE ON THE RATE OF DESORPTION OF TRITIUM ABSORBED ON STAINLESS STEEL

DURATION / TEMPERATURE	2 HRS (100°C)	4 HRS (200°C)	6 HRS (350°C)	8 HRS (1000°C)
BUBBLER B1 (mCi)	1.02	2.3	3.1	3.6
BUBBLER B2 (mCi)	0.002	0.012	0.021	0.025
TOTAL HTO (mCi)	1.022	2.312	3.121	3.625
BUBBLER B3 (uCi)	1.75	6.29	46.1	108.3
BUBBLER B4 (uCi)	0.0002	0.004	0.018	0.132
TOTAL HT (uCi)	1.7502	6.294	46.118	108.432

Figures 2 and 3 plot the HTO and HT released respectively expressed as a percentage of the total. At 100°C only 20% of the total tritium desorbed over a 2 hour period. There was an initial rapid release of tritium as HTO in the first 30 minutes but then the rate of desorption decreased to 3.8% per hour. At this temperature essentially all of the tritium desorbed as HTO. As the temperature was increased to 200°C the rate of desorption of tritium increased to 22% per hour with HTO being the predominant desorbing species. At the end of the two hour period at this temperature, 80% of the total tritium, as HTO, had been released. Between 350°C and a 1000°C the remaining 20% of tritium desorbed over a 4 hour period. The total amount of HT desorbed was 3% of the total and most of this was released when the temperature was increased to 350°C.

This experiment established that relatively low temperatures are able to thermally decontaminate stainless steel in a matter of a few hours.

In the second experiment, a second tubing sample was heated at 350°C for 4 hours and subsequently for a further 4 hours at 1000°C. Figures 4 and 5 show the cumulative tritium released as HTO and HT as a function of time. The total tritium removed after 8 hours was 6 mCi which represents a surface activity of 0.33 mCi/cm^2 in excellent agreement with the previous measurement. Within the first hour, 70% of the total tritium was released and after 2 hours, 92% of the total tritium had been removed. As in the previous experiment the predominant species was HTO (95%). Increasing the temperature to 1000°C did not have any noticeable impact on the tritium desorption rate in this experiment because the quantity of tritium remaining in or on the metal was negligible when the temperature was increased.

Finally in the third experiment a 3 cm length of the tubing was placed into the tube furnace at 1000°C to simulate the instantaneous heating that would occur during a welding operation. This was considerably lower than the temperature attained at the torch tip during welding but it was the maximum temperature we could obtain with our equipment. Assuming that the volume of the system was approximately $500cm^3$ and that all of the tritium is released instantaneously, the expected reading on a high range Scintrex connected to the outlet of the tube furnace was 7.8 Ci/m^3. The instrument registered a peak of 6.5 Ci/m^3 within 30 seconds confirming that essentially all of the tritium inventory in the heated zone would be instantaneously released during welding.

CONCLUSIONS AND RECOMMENDATIONS

Based on two separate determinations the surface tritium activity of the tubing obtained from the Darlington Tritium Removal Facility is 0.33 mCi/cm^2. The predominant tritium species to be released is HTO. The HT component is less than 5% of the total. The quantity of smearable tritium contamination (1.3 mCi) was 26% of the total tritium activity.

Thermal desorption of tritium at 350°C was an effective technique for the decontamination of surface tritium from stainless steel surfaces. This temperature is not expected to adversely affect the ability to weld the tubing.

The airborne tritium concentration in an unventilated 2 m^3 plastic tent generated by cutting, and then heating a 30 cm length of tubing is approximately 2500 MPC_a assuming an MPC_a value of 10 uCi/m^3 for HTO and also assuming that all of the desorbed tritium escapes into the tent atmosphere. Clearly, good ventilation of the tent atmosphere is of paramount importance.

The tubing used for this work was contaminated on the outside as well as on its internal surface. The calculations in fact assume a uniform distribution of tritium over the entire tube surface both inside and out. The actual field tubing to be modified is contaminated only on the inside. Therefore there is some benefit in preheating the tubing over the length that will be affected by welding and purging the tubing with an inert gas to reduce the tritium inventory in preparation for cutting and welding of the tube. It is very probable however that recontamination of the cleaned area will take place as purge gas picks up tritium and deposits it onto clean surfaces. This work did not address the recontamination issue but it would be prudent to assume that the rate is rapid. Consequently, if preheating of the tubing is used as a decontamination technique it should be applied just before tube cutting in order to minimize the potential for recontamination.

The experiments carried out in this work were designed to answer very specific concerns regarding tritium surface contamination required to be resolved for work planning purposes. In order to develop effective decontamination techniques for a variety of expected tritium contaminated surfaces it will be necessary to undertake additional systematic studies to:

(a) characterize the nature of tritium surface contamination on different surfaces, metallic as well as non metallic including different grades of stainless steel as a function of tritium partial pressure and exposure time,

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(d) to assess the rate of recontamination of a decontaminated surface in both a static and flowing gas environment.

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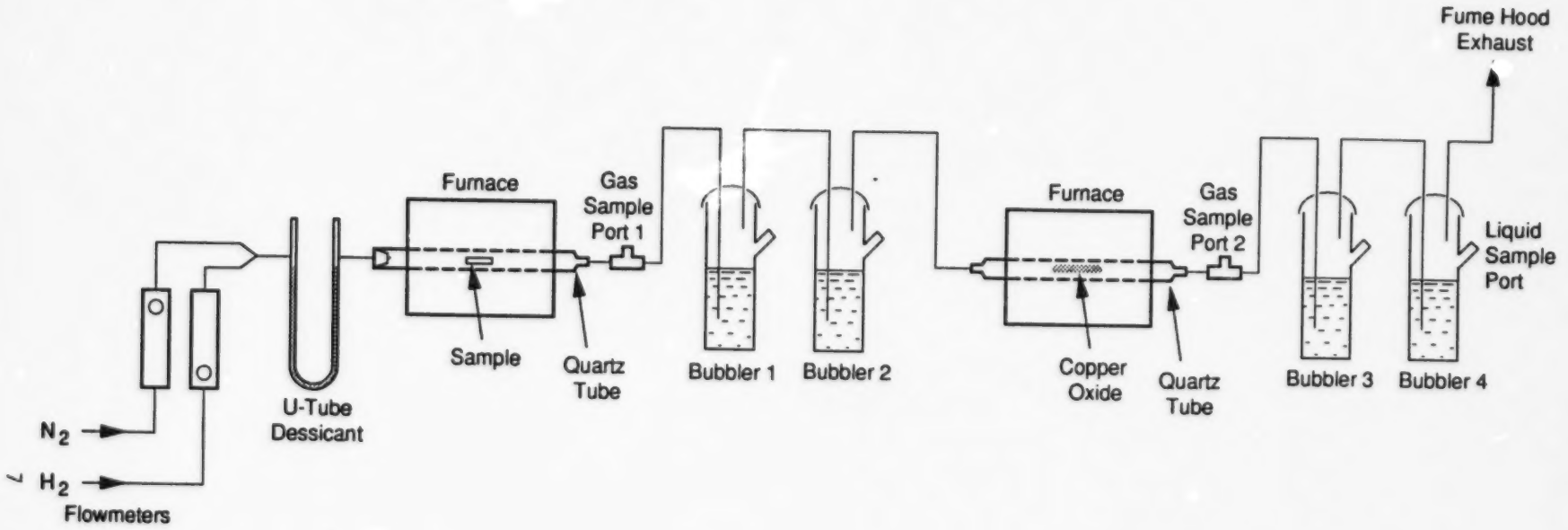

FIGURE 1

TRITIUM DESORPTION EQUIPMENT SCHEMATIC

Figure 2: HTO Release from Stainless Steel Sample #1

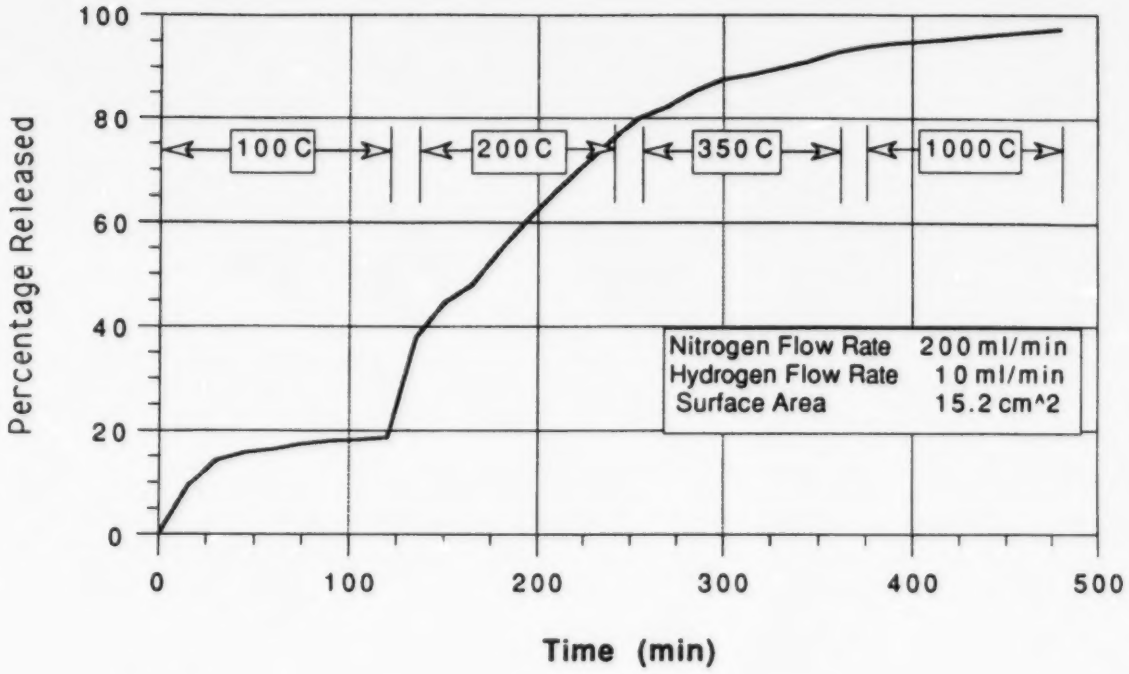

Figure 3: HT Release from Stainless Steel Sample #1

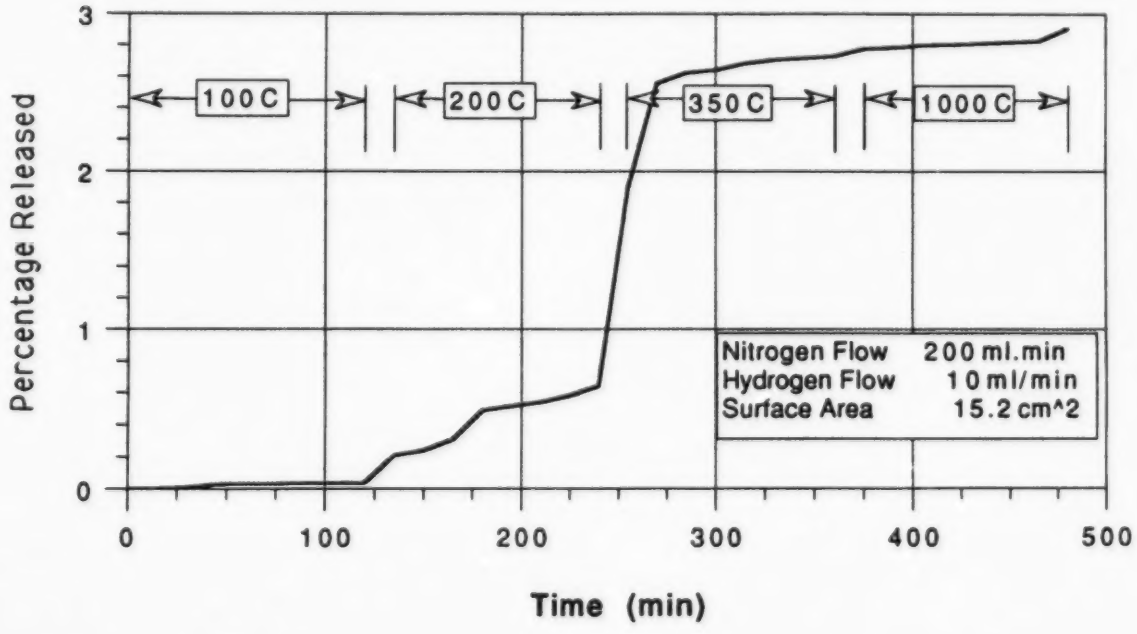

Figure 4: HTO Release from Stainless Steel Sample #2

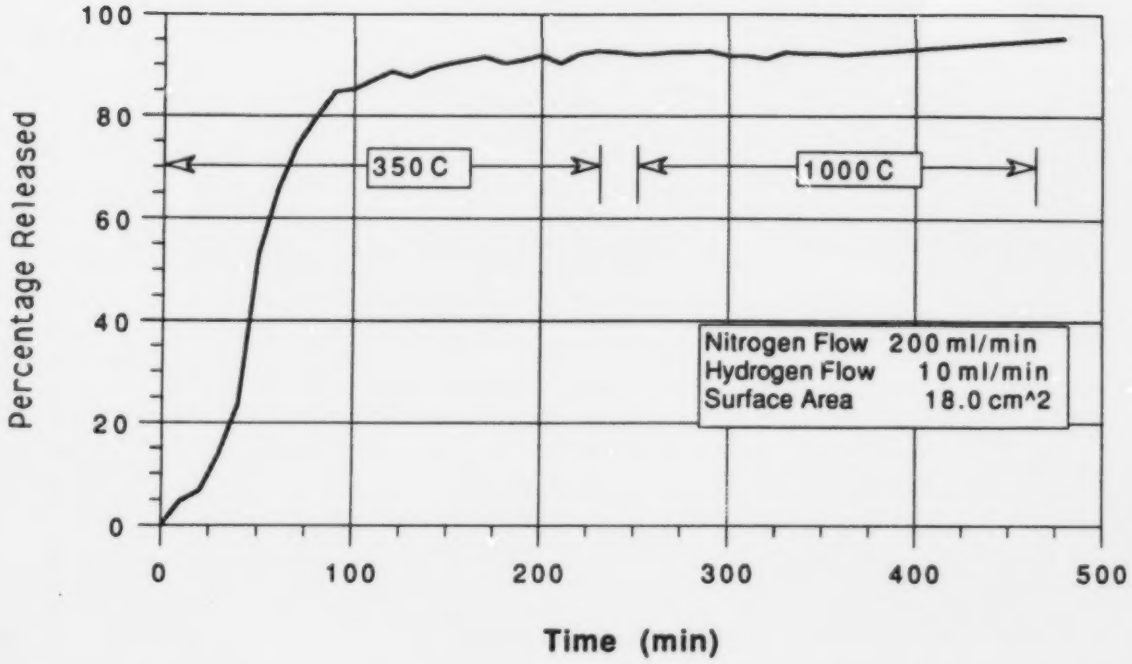

Figure 5: HT Release from Stainless Steel Sample #2